Agilent G1732BA MSD Security ChemStation

User's Guide

Agilent Technologies

Notices

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Acknowledgements

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Software Revision

This manual is valid for B.02.xx revisions of the Agilent G1732BA MSD Security ChemStation software, where xx refers to minor revisions of the software that do not affect the technical accuracy of this manual.

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WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

In this guide...

This manual contains condensed information that is often needed by MSD Security ChemStation users.

1 Introduction

This chapter presents a brief description of the software.

2 Security Panels

This chapter displays the security panels used with the software.

3 Online Help

This chapter describes the controls and icons for online Help.

4 Instrument Control

This chapter describes the controls that set and monitor instrument parameters.

5 Procedures

This chapter contains a number of frequently used procedures, both hardware and software.

6 Tips

This chapter contains brief descriptions of operation and troubleshooting tips.

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1 Introduction

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NOTE The G1732BA MSD Security ChemStation is software that should be installed on a computer dedicated to this specific application. This software supports local LAN acquisition for one GC/MSD system. Other applications and/or wide area network access should be performed on other computers.

This guide is a collection of information about the system that has proven useful to other users. Use the online Help capability, described in Chapter 2, for additional information.

The Agilent G1732BA MSD Security ChemStation and the G1742BA MSD Security ChemStation Upgrade are designed to support the requirements of the U.S. Food and Drug Administration (FDA) regulations on the handling of electronic records and electronic signatures published as 21 CFR Part 11.

The systems, hereafter referred to as the MSD Security ChemStation, acquire data from one Agilent Mass Selective Detector (MSD) with an Agilent 6850, 6890, or 7890A Series Gas Chromatograph (GC).

The MSD Security ChemStation

The MSD Security ChemStation is a conventional MSD ChemStation with support for U.S. Food and Drug Administration (FDA) 21 CFR Part 11 regulations. These regulations govern the use of electronic records, electronic signatures, and auditability of systems used in regulated laboratories.

Capabilities and limitations

The MSD Security ChemStation supports the same hardware as the standard MSD Productivity ChemStation. However, it is focused on GC/MSD signal acquisition and will acquire GC detector data only in combination with GC/MSD data acquisition. It will also import and process existing GC/MSD files that contain GC data.

2
Security Panels

The MSD Security Monitor Panel 12
The Login Panel 13
The Signature Panel 14

These panels are used throughout the MSD Security ChemStation software.

The MSD Security Monitor Panel

This monitor appears in the lower right corner of the screen after installation (Figure 1).

Figure 1 The MSD Security Monitor

It can be moved to other positions but must remain visible whenever the MSD Security ChemStation software is in use. If it is not present, restart it using **Start/All Programs/MSD Security ChemStation/MSD Security Monitor**.

The monitor displays the Full Name of the current user, as required by the FDA regulations. It also provides these functions:

- **Change Password**—Used at first login to set the user password, and then to periodically change the password. The rules for setting up your password are determined by the Windows® password policy. You can also access these rules by checking Windows Help or by contacting your local system administrator.
- **Lock Session**—Locks the session until an authorized user unlocks it. This is typically used when operators change while the system is running, for example, during shift changes.
- **Private Lock**—By checking this function and pressing **Lock Session**, the session is locked until the current user or a Manager unlocks it.
- **Help**—Opens the online Help.

The Login Panel

This panel (Figure 2) appears when a user attempts to run Instrument Control or Data Analysis.

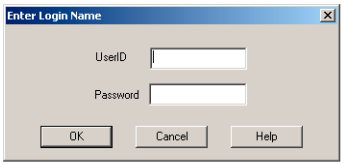

Figure 2 Login panel

The MSD Security ChemStation requires all users to sign in to the system using their previously-designated UserID. To sign in to the MSD Security ChemStation, enter your UserID and password into the appropriate fields and click **OK**.

NOTE Your UserID will be locked out of the system after five failed login attempts. If you are having trouble logging in, see your system administrator for help.

The Signature Panel

To ensure a secure system, the MSD Security ChemStation stamps saved items and generated results (such as methods, sequences, and quantitation results) with an electronic signature. This signature notes the current user, date, time of the event, and any pertinent comments. The Signature Panel (Figure 3) will appear automatically when an electronic signature is required.

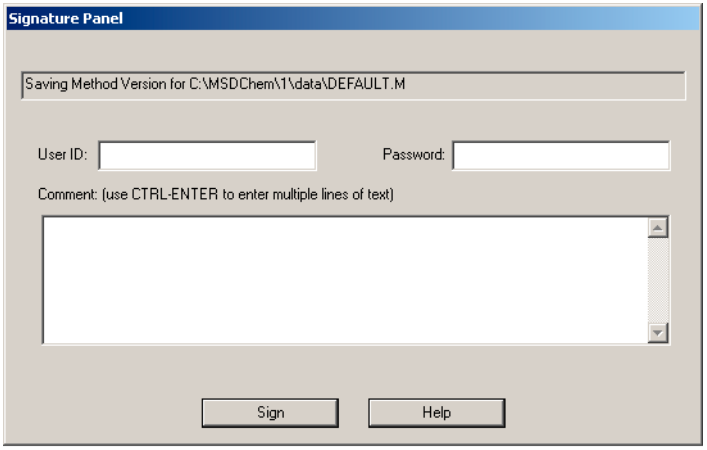

Figure 3 Signature panel

The ChemStation logs these signature entries for auditing purposes.

After entering the User ID and password to confirm identity, the user *must* enter a comment. This comment becomes a permanent part of the audit trail. We recommend that it be a brief note explaining why the information is being saved.

If the Lab Manager (or Administrator) has decided to turn the electronic signature off, the operator will not be prompted for signatures when results are made or a method is saved. If desired, the user can still use the **Manually Sign Method** or **Manually Sign Data** options under the **Tools** menu in **Data Analysis** to record an electronic signature and comment.

3
Online Help

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Use online Help to learn about instrument control, data acquisition, data analysis, methods, sequencing, and tuning.

Help Controls

To access online Help, select **Help Topics** from the Help menu in any window or click **Help** on any dialog box. See Table 1 for the available controls.

Table 1 Help controls

Item	Description
Hide/Show	Turns on or off the list of Help topics
Back	Navigates back to the previously viewed Help topic
Forward	Navigates to the next Help topic
Print	Prints the current book or Help topic
Contents	Displays the list of Help topics
Index	Searchs the Help index for a particular topic using your keywords
Search	Displays a list of all the topics in the online Help that contains your keyword or phrase

Help Icons

The icons shown in Table 2 appear in some of the Help topic listings.

Table 2 Help icons

	Indicates a book containing more Help topics. To open a book, select it, then double-click.
	Indicates an open book of Help topics. To close an open book, select it, then double-click.
	Indicates a Help topic. To jump to a Help topic, select it, then double-click.

3 Online Help

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Instrument Control

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The Instrument Control view is used to set and monitor instrument parameters.

Instrument Control View

The Instrument Control view (Figure 4) is displayed when you start up the MSD Security ChemStation. If you are in a different view, select **View/Instrument Control** when you are ready to set up the instrument for data acquisition.

See the online Help for more details on the menus, buttons (Table 3), or windows used in the MSD Security ChemStation software.

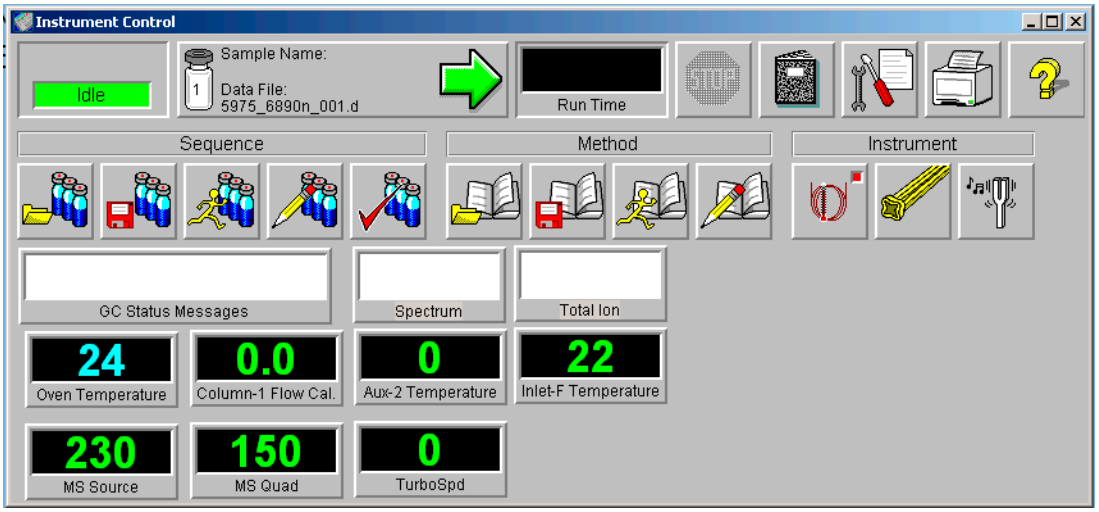


Figure 4 Instrument Control view

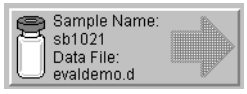
Instrument Control Buttons

The buttons that appear in the Instrument Control view are described in Table 3.

Table 3 Instrument Control buttons

Acquisition Status Indicator
Displays the status of the current run.

Run Time
Displays the remaining time in a run.



Start Run
Displays the sample name and data file ready to run.

Stop
The stop sign is red during PreRun, Run, or PostRun, and gray when a run is not in progress. Use this button to stop the system when it is in PreRun, Run, or PostRun. If the system is in Run, the system will go to PostRun. If the system is in PostRun, it will go to Idle.

Logbook
Displays the logbook popup menu.

Maintenance Due
Displays the Select Early Maintenance Feedback (EMF) Action dialog box.

Print
Displays a dialog box with printable items such as sequence log, current sequence, instrument parameters, Data Analysis parameters, and detailed Data Analysis parameters.

Help
Displays Help for the Instrument Control view and gives access to the complete Help system.

Table 3 Instrument Control buttons (continued)

	Load Sequence Opens the Load Sequence dialog box.
	Save Sequence Opens the Save Sequence dialog box.
	Run Sequence Opens the Start Sequence dialog box.
	Edit Sequence Opens the Sample Log Table dialog box.
	Simulate Sequence Tests a sequence.
	Load Method Opens the Load Method dialog box.
	Save Method Saves the current method.
	Run Method Opens the Start Run dialog box.
	Edit Method Edits the current method.
	GC Parameters Edits the GC parameters and GC monitors.
	MS Parameters Edits the MS parameters and MS monitors.
	Tune Parameters Tunes the MSD.

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This chapter contains the commonly-used procedures for the GC/MSD system.

To Set a Password

New users log in to the MSD Security Chemstation using a User ID and password designated by their Windows Administrator.

After this first log on, and then periodically thereafter, users are required to change their password.

To change your password:

1 From the MSD Security Monitor, select **Change Password**. (see "The MSD Security Monitor Panel"on page 12.) The **Change Password** dialog box appears.

2 Enter the required information in the space provided. See your Windows Administrator for password rules.

3 Select **OK**.

To Pump Down (Start Up) the MSD

1 Make sure your system meets all of the following conditions before you pump down:

- ❑ The vent valve is closed (the knob is turned all the way clockwise).
- ❑ All other vacuum seals and fittings are in place and fastened correctly. (The front side plate screw *should not be tightened.*)
- ❑ The MSD is connected to a grounded power source.
- ❑ The GC/MSD interface extends into the GC oven.
- ❑ A conditioned capillary column is installed in the GC inlet and in the GC/MSD interface.
- ❑ The GC is on, but the heated zones for the GC/MSD interface, the injection port, and the oven are off.
- ❑ Carrier gas of at least 99.999% purity is plumbed to the GC with the recommended traps.
- ❑ If hydrogen is used as carrier gas, carrier gas flow is off and the front sideplate thumbscrew is loosely fastened.
- ❑ The foreline pump exhaust is properly vented.

WARNING **Make sure your MSD meets ALL the conditions listed above. Failure to do so can result in personal injury.**

2 From the Instrument Control view, select **Tune and Vacuum Control/Pump Down**.

Note: Analysts and managers in the Tune and Vacuum Control view can select **Vacuum/Pump Down**.

3 When prompted, switch on the MSD.

4 Press lightly on the side board to ensure a correct seal.

The foreline pump will make a gurgling noise. This noise should stop within a minute. If the noise continues, there is a *large* air leak in your system, probably at the side plate seal, the interface column nut, or the vent valve.

5 Once communication with the MSD is established, click **OK**. Within 10 to 15 minutes the diffusion pump should be hot, or the turbo pump speed up to 80%. The turbo pump should eventually reach 95%.

CAUTION If these conditions are not met, the foreline pump will be shut off. You must then power cycle the MSD. If the MSD does not pump down correctly, see the online Help for information on troubleshooting air leaks and other vacuum problems.

6 When prompted, turn on the GC/MSD interface heater and GC oven. Click **OK** when you have done so. The software will turn on the ion source and mass filter (quad) heaters. The temperature setpoints are stored in the current autotune (*.u) file.

CAUTION Do not turn on any GC heated zones until carrier gas flow is on. Heating a column with no carrier gas flow will damage the column.

7 After the **OK to run** message appears, wait 2 hours for the MSD to reach thermal equilibrium.

CAUTION Data acquired before the MSD has reached thermal equilibrium might not be reproducible.

CAUTION If using a toxic gas, for example ammonia, tighten the MSD side plate screws. Tightening these screws before reaching vacuum can distort the seal and cause leakage.

To Vent (Shut Down) the MSD

1 If your 5975 Series MSD is equipped with a vacuum gauge controller, from the Tune and Vacuum Control view select **Vacuum/Turn Vacuum Gauge on/off.** Make sure the external Ion Gauge Controller for the 5973 Series MSD is turned off.

2 Turn the gauge off.

3 Before venting a 5973 series CI MSD, press [**Gas Off**]. This turns off the reagent gas flow and closes the isolation valve.

WARNING **On a 5973 CI MSD, the Gas Off light must be on when the MSD is venting.**

4 From the Instrument Control view, select **Instrument/Vent**. Follow the instructions presented.

Note: Analysts and managers in the Tune and Vacuum Control view can select **Vacuum/Vent**.

5 Set the GC/MSD interface heater and the GC oven temperatures to ambient (25 °C).

WARNING **If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MSD power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read the Hydrogen Carrier Gas Safety Guide (5955-5398) before operating the MSD with hydrogen carrier gas.**

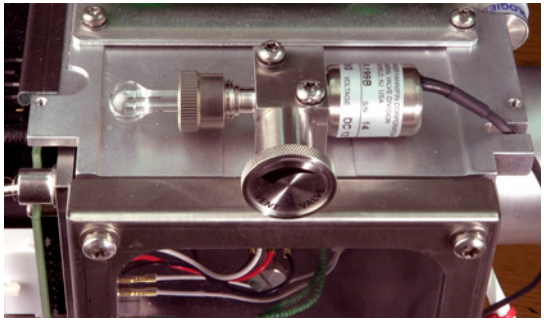
CAUTION Be sure the GC oven and GC/MSD interface are cool before turning off carrier gas flow.

6 When prompted, turn off the MSD power switch.

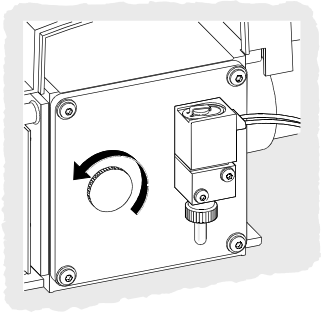
7 Unplug the MSD power cord.

8 Remove the analyzer cover (5973 series) or the source view window cover (5975 series).

9 Turn the vent valve knob counterclockwise **only** 3/4 turns or until you hear the hissing sound of air flowing into the analyzer chamber.



5975 Vent valve



5973 Vent valve

Figure 5 Venting the 5973 analyzer

CAUTION Do not turn the knob too far, or the O-ring may fall out of its groove. Be sure to retighten the knob before pumping down.

WARNING **Allow the analyzer to cool to near room temperature before touching it.**

CAUTION Always wear clean gloves while handling any parts that go inside the analyzer chamber.

To Tune the MSD

You should tune the MSD periodically to maintain its optimum performance. Tuning is the process of adjusting MSD parameters so the instrument meets certain performance criteria. How often you should tune is determined by the number and type of samples you are running, as well as the overall condition of your system.

Always tune the MSD with the GC oven temperature, column flow, and analyzer temperature that will be used for data acquisition.

Keep the Tune reports in a notebook so that successive reports can be easily compared.

From the Instrument Control view:

1 Select the **Tune Parameters** icon (displays only the first two menus listed in step 2) or **View/Tune and Vacuum Control**.

2 From the **Tune** menu select one of the following, depending on the instrument performance required by your application:

- **Tune MSD**
  Results in maximum sensitivity over the full scan range.
- **QuickTune**
  Adjusts the peak width, mass assignment, and abundance without changing ion ratios.
- **Autotune (Atune.U)**
  Tunes for maximum response over full scan range.
- **Low Mass Autotune (Lomass.U)**
  Tunes for the low-mass range.
- **Standard Spectra Tune (Stune.U)**
  Results in a standard response over the full scan range. This option may reduce sensitivity.
- **Tune Wizard. . .**
  Displays a series of dialog boxes that let you set abundance ratio targets and adjust tuning criteria. This is used for target tuning.

- **Air and Water Check**
  Generates a standardized measurement and report of the system air (nitrogen *m/z* 28) and water (*m/z* 18) levels relative to PFTBA mass 69. Use this item to check for leaks. The abundance of *m/z* 28 should be less than that of *m/z* 18, and each should be less than 5% of *m/z* 69.

- **Tune Evaluation**
  Evaluates the current tune file.

3 Review the Tune report.

4 To view the history of tune results, select **Tune/View Tunes**.

To Use Manual Tune

Manual tuning lets you interactively set the MSD parameters, such as lens voltages and tuning masses, to values that meet the needs of your particular analysis. You can often obtain greater sensitivity with manual tuning than you can with autotuning.

Manual tuning allows you to ramp individual parameters and to specify the range and step size for the ramp. The results of the ramp are displayed visually with the optimum value for the parameter clearly marked on the plot.

You can acquire two types of data in manual tune: profile scans (plots the abundance and peak shape of the tune masses) and spectrum scans (scans plot response across the entire mass range).

See the online Help for more details about manual tuning.

To Acquire Data

To set up the GC for use with the MSD

In the Instrument Control view:

1. Select **Instrument/Inlet/Injection Types.** Select the appropriate injection source and then select the **Use MS** checkbox. Click **OK**.
2. Select **Instrument/Edit GC Parameters**.
3. Click **Aux**. Verify that you are using auxiliary channel 2, the heater is on and set to the desired temperature, and that *MSD* is selected as the Type.
4. Click **Columns**. Verify that the detector is *MSD* and that *Vacuum* is selected for Outlet psi. Click **OK**.

To inject a sample with the autosampler

In the Instrument Control view:

1. Place the autosampler vial containing the sample into the autosampler tray.
2. Click the **Run Method** icon or select **Method/Run Method**.
3. When the **Start Run** box appears, specify the sample information:
   - ❏ Specify a unique data path for the sample.
   - ❏ Specify a unique data file name for the sample.
   - ❏ Enter the position number of the sample vial in the **Vial** field (1–100).
   - ❏ (Optional) Fill in the **Sample Name** and **Misc Info** fields to document the injection.
   - ❏ Make sure that the **Data Acquisition** option is selected. Select the **Data Analysis** option if you want to generate any of the reports specified in the method.
4. Click **Run Method** to initiate the run.

CAUTION Do *not* use **Start** on the GC to start a run when using the autosampler.

To inject a sample manually

In the Instrument Control view:

1 Select **Instrument/Inlet/Injector Types**.

2 In the **Inlet and Injection Parameters** dialog box, select **Manual** as the injection source.

3 On the GC keypad, press [**Prep Run**]. This cancels the gas saver flow, brings the inlet flow to its setpoint value, and closes the purge valve (for splitless injection only).

4 Select **Method/Run Method**.

5 When the **Start Run** box appears, specify the sample information as described below:

- ❏ Specify a unique data path for the sample.
- ❏ Specify a unique data file name for the sample.
- ❏ (Optional) Fill in the **Sample Name** and **Misc Info** fields to document the injection.
- ❏ Make sure that the **Data Acquisition** option is selected.
- ❏ (Optional) Select the **Data Analysis** option if you want to generate any Data Analysis reports specified in the method.

6 Click **Run Method** to initiate the run. If the temperatures are stable, the **Prepare To Inject** box appears. Otherwise, the message **Waiting for GC ready** is displayed.

7 When the GC temperatures have stabilized (6890 GC - the Pre Run light on the GC is steady, 6850 GC - the Not Ready light is off), inject the sample and press [Start] on the GC keypad.

CAUTION Do not inject before the GC is ready. This will cause inconsistent results.

To Analyze MS Data

You can load a data file from the **Navigation panel** or by selecting **Load Data File** in Data Analysis view.

To load a data file

To load the data file in Data Analysis:

1 Select the **Load Data File** icon or select **File/Load Data File**.

2 Select a data file (double-click on a file name, or type a name, and click **OK**). The chromatogram for the data file is loaded and displayed in window [2].

CAUTION A data file must be loaded to perform any of the tasks in this section.

To integrate a chromatogram

1 Select **Chromatogram/Integrate**.

2 After providing an electronic signature, select **Chromatogram/Percent Report** to see results.

To select a spectrum

NOTE If right-clicking the mouse in window [1] or [2] displays a menu, use the **Switch Data Analysis Mouse Actions** button to toggle between right-click modes.

Double-click the *right* mouse button on the time point of interest in the chromatogram. The spectrum appears in window [1].

To zoom in

1. Position the pointer at one corner of the area you wish to expand in a chromatogram or spectrum.
2. Press and hold the *left* mouse button while dragging the mouse to select the area you wish to expand.
3. Release the mouse button. The selected area expands to fill the existing window.

To zoom out

1. Position the mouse anywhere in the zoomed window.
2. Double-click the *left* mouse button.

To average spectra

1. Position the pointer in the chromatogram at the starting time for the range you want to average.
2. Press the *right* mouse button while dragging the mouse to the end of the range you want to average.
3. Release the mouse button. The spectra in the selected range are averaged and the averaged spectrum is displayed in window [1].

To add two spectra

1. Select a spectrum (double-click the *right* mouse button in the chromatogram).
2. Select a second spectrum (double-click the *right* mouse button in the chromatogram).
3. Select **Spectrum/Add**. The two spectra are added together and the resulting spectrum is displayed in window [1].

To subtract two spectra

1. Select a spectrum (double-click the *right* mouse button in the chromatogram).
2. Select the spectrum to be subtracted (double-click the *right* mouse button in the chromatogram).
3. Select **Spectrum/Subtract**.

The spectrum selected in step 2 is subtracted from the spectrum selected in step 1 and the resulting spectrum is displayed in window [1].

To subtract background spectra

1. Select a spectrum or average a range of spectra to subtract from the data file.
2. Select **File/Subtract Background (BSB)**. The system performs the following tasks:
   - The selected spectrum is subtracted from every scan in the current data file.
   - The subtracted data is stored in a BSB subdirectory in the same directory as the data file.

The subtracted data file becomes the current data file and is displayed in window [2].

To Save a Method

A method saved in the **\METHODS** directory is protected from changes by **Operators**. Only **Managers** and **Analysts** can save methods in this directory and must explicitly specify it as the save location.

Example: Adding a Quant database to a method using AutoQuant Setup

Consider adding a quant database to a method (for details on each step, see the Help system).

1. In Data Analysis, load the method and data file.
2. Select **Calibrate/AutoQuant Setup**.
3. Enter information and compound data as appropriate.
4. When prompted to **Quantitate now?**, select **Yes**. As quantitation proceeds, the signature panel appears. The sign panel shows that the quantitated method version will be saved under the *loaded data file's* path.
5. Complete the tasks required by the **Edit Compounds** dialog box.

At this point, the quant database has been added to the method. However, note that the method version was saved under the *loaded data file's* path. To save the quantitated method as a protected method in the **\METHODS** directory, select **Method/Save method...** and browse to **\METHODS** directory.

To View Previously-Generated Reports

For full traceability, the MSD Security ChemStation retains all result versions by applying strict revision control of all records maintained by the system. A time stamp uniquely identifies each version.

Each report that is generated is stored as an electronic record. All result versions and the reports associated with them can be easily reviewed in the Review Result Versions Viewer. The viewer provides a list of all result versions of an electronic record in chronological order.

To view, print, or export this information:

1. Log on to Data Analysis.
2. Load the data file you want to review.
3. Select **Tools/Review Result Versions**. The Result Versions Viewer opens.
4. On the left side of the screen (Electronic Record), select the version of the data file you want to review. The reports associated with the selected version are displayed on the right side of the screen (Reports in Electronic Record).
5. On the right side (Reports in Electronic Record), select the report you want to view. The content of the report is displayed in the Report section of the screen.
6. Perform optional actions.
   a. Print sends only the selected report to the default printer.
   b. Export sends all reports associated to the selected version of the data file to a specified directory on your PC.
   c. Sign allows you to add a new electronic signature to the data file for auditing purposes.
7. Click **OK** to return to Data Analysis.

To Set the Private Lock

1 Mark the **Private Lock** checkbox, and then click the **Lock Session** button, in the **MSD Security Monitor**.

Figure 6 Private Lock checkbox

The **Session Lock** dialog box is displayed, and the MSD Security ChemStation session is locked. It can only be unlocked by the named user or by a manager.

NOTE The **MSD Security Monitor** and **Session Lock** dialog boxes do not display the **User ID**, but the full name of the user who signed in.

2 In the **Session Lock** dialog box, type **Analyst** in the **UserID** field, and **CS03security** in the **Password** field. Remember that the login is case-sensitive.

A **Login Failed** error is displayed, because when the MSD Security ChemStation is privately locked, only the named user or a **Manager** can unlock it.

Private Lock is for those occasions when the correct user has to leave the ChemStation for a short time, for example, for a break.

3 Repeat the login using the correct **UserID** and **Password**, as in step 2. The screen is no longer dimmed so the applications are again accessible.

To Use Spectral Libraries

To select a library

In Data Analysis, select **Spectrum/Select Library**.

In the **Library Search Parameters** dialog box, enter the name of the Library on the first line.

Up to two additional Search libraries may be entered. Searching in these additional libraries is dependent on a compound being found that meets the match quality specified.

To integrate and search peaks

Use the following procedure to integrate a total ion chromatogram (TIC) and automatically generate a library search report for each peak detected.

1 In Data Analysis, load a data file. The TIC is displayed.

2 Select **Spectrum/Library Search Report**.

3 When the **Library Search Report Options** dialog box appears, select the options you want for the library search report:

- Select either **Summary** or **Detailed** to determine the report format.
- Select one or more destinations (**Screen**, **Printer**, and **File**).
- Select an **Integration Parameter File** (leave the field blank to autointegrate using the GC/MSD Security ChemStation integrator).
- Select which spectrum from each peak to use (**Apex**, **Apex - Start of Peak**, **Apex - Background at time**, or **Peak Average)**.

4 Click **OK** to initiate the search. The Signature Panel is displayed.

The chromatogram is integrated and a spectrum from each peak is searched. The results of the integration appear on the screen. The library search report is sent to the destinations selected in step 3.

To search a single spectrum

1 In Data Analysis, load a data file. The TIC is displayed.

2 Select a spectrum by double-clicking the right mouse button on any spectrum to display it in a window below the chromatogram.

3 Initiate the library search by double-clicking the right mouse button in the window containing the spectrum.

NOTE If right-clicking the mouse in window [1] or [2] displays a menu, use the **Switch Data Analysis Mouse Actions** button to toggle between right-click modes.

4 When the search is complete, the search results appear on the screen. The spectrum for the unknown, the reference spectrum you select from the list of hits, and, if available, the chemical structure of the reference compound is displayed.

5 To view other spectral data:

   a Click on another compound in the hit list to display a different reference spectrum.

   b Select **Difference** to display the difference between the unknown and the reference spectra.

6 To view other information:

   a Click **Statistics** to display information about the quality of each hit found in the list.

   b Click **Text** to view the header information stored in the library for the current reference spectrum.

7 Click **Print** to print a copy of the displayed spectra.

8 Click **Done** to clear the library search results from the screen.

To Reprocess a Sequence

Reprocessing runs independently of the online instrument, so reprocessing a previously-run sequence can occur while collecting new data. It can also reprocess a sequence using an edited Sample Log Table, such as one containing different sample amounts. When the modified sequence is run, any revised file versions are saved under the sequence's run directory path.

To Set the Time-Based Session Lock

1 Right-click anywhere on the desktop and select **Properties** from the **Context** menu. In the **Display Properties** dialog box, select the **Screen Saver tab**.

The time-based session lock is managed by the special Agilent GCMS 21CFR11 screen saver.

2 Change the Wait time to 1 minute and click **OK** to close the **Display Properties** dialog box.

3 Wait for 1 minute. The **Session Lock** dialog box is displayed, and the workstation is locked.

4 Enter the appropriate **UserID** and **Password** to unlock the workstation, then open the **Display Properties** dialog box again and reset the Wait time to its previous value.

NOTE You cannot access the **Settings**, or see a **Preview** of the Agilent GCMS 21CFR11 screen saver.

5 Click **OK** to close the **Display Properties** dialog box.

NOTE Setting the Screen Saver to *None* will not work and is not supported.

To Import and Export Data, and Method Files

You must be a member of the MSDManager or MSDAnalyst Group to perform these tasks.

Import

Import can copy a **.m** (method), **.s** (sequence), or **.d** (data) file from an external medium such as a CD-ROM or floppy disk into the MSD Security ChemStation.

Export

Export can copy a **.m** (method), **.s** (sequence), or **.d** (data) file from the secure file system to either the hard disk or an external device. The exported files do not include the security restrictions or audit details from the original files. The original files are not changed.

To Setup a Data Path

The MSD Security ChemStation allows for using a predefined directory structure to contain run directories, or a user-defined structure. To select where run data directories should be created, open the **Instrument** view and the **Method** menu. Select **Set New Data Paths. . .** A dialog box appears (Figure 7):

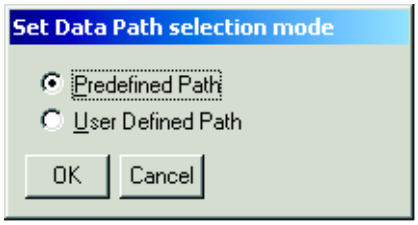


Figure 7 Set Data Path selection mode dialog box

Select the desired option.

Predefined path structure

In this mode, the predefined directory structure for data is shown in Figure 8.

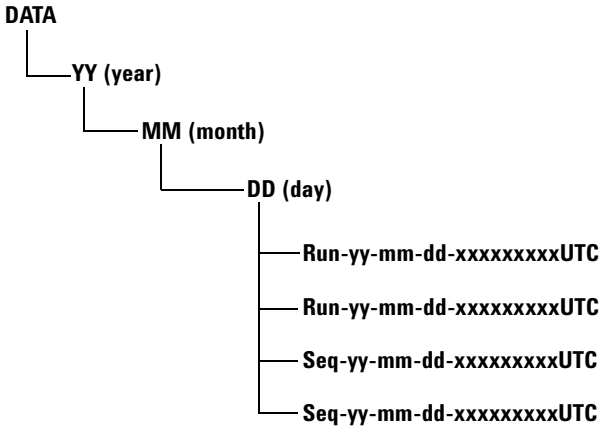


Figure 8 Predefined data structure

This structure insures that no single folder is overwritten with data files, and the use of Year\Month\Day is closely associated with the naming convention of the actual run and sequence subfolders.

If this predefined path structure is used, the **Browse** button on the **Start Run** and **Start Sequence** panels is disabled. The Data File Directory (Data Path) text box is inactive and displays a message indicating the predefined structure that will be used.

User-defined path structure

Users can design their own structure below the **..\DATA** directory. This allows a user to group data by product (or any other criterion) rather than strictly by date.

To set up a user-defined structure, select the **User Defined Path** option from the **Set Data Path selection mode** dialog box (Figure 7 on page 45) and click **OK**. The **Select Path for Data Files** dialog box appears (Figure 9):

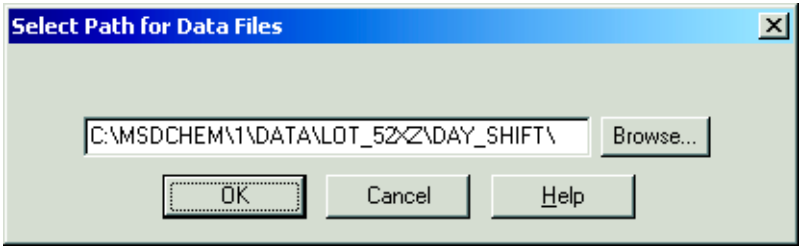


Figure 9 Select Path for Data Files dialog box

Use this dialog box to type in or **Browse** to a directory path, then select **OK**.

- New directories and subdirectories can be created.
- All user-created directories are browsable when running sequences and methods, so multiple directories can be created in advance.

There are some limitations upon the paths that a user can specify.

- Must be a subfolder to **systemdirectory\....\DATA**
- Folder name cannot begin with **Run-**
- Folder name cannot begin with **Seq-**
- Folder name cannot begin with **Imp-**

When a user-defined path is in use, the **Browse** button and the **Data File Directory/Data Path** text fields are active in the **Start Sequence** and **Start Run** panels.

To return to the predefined data directory structure, go back to the **Set Data Path selection mode** dialog box. Select **Predefined Path** and **OK**.

To Archive and Restore MSD Security ChemStation Data

You must be a member of the MSDManager or MSDAnalyst Group to perform these tasks.

Archive and restore

Archiving provides a means of transferring data (and associated methods and sequences) to an external medium to free-up hard disk space. Restore brings previously archived information back into the system. Archives can be restored to either the original MSD Security ChemStation installation or to another system on another PC.

All archiving activities are documented in the system audit trail. Also, an archive catalog, <archivename>.MANIFEST, is generated for each archive unit. See page 49 for procedures to create and restore an archive.

Archiving can be used to transfer a copy of all data (plus associated methods, sequences, and accompanying audit information, including electronic signatures) to:

- A location on your hard disk outside the MSDChem directory
- A DVD/CD

It is NOT possible to archive files from the MSD Security software directly to a networked drive.

The data archival tool allows archives to be created with or without deletion of the data stored in the MSD Security ChemStation.

XML catalog file

Each time archive is run, the system generates a catalog file (<archive name>.MANIFEST) in XML format for each archived unit. This file describes the content of the archive unit on a sample by sample level. It can be used, for example, for data handling in archive management or knowledge engineering systems. All archiving activities are documented in the system audit trail.

Procedure to create an archive

To create an archive:

1 Start **Data Analysis**.

   a On the desktop, double-click **Data Analysis**.

   b Log on.

2 Set up the archive.

   a Select the **Archive/Restore** menu.

   b Select a menu item.

   c Select a directory on the local hard drive for the archive.

   d Select the specific items to be archived. Use the **Select** button. When done, click **OK**.

   e Specify whether to delete (normal behavior to recover space) or not delete (for sending data to a regulatory body) items after archiving.

NOTE

If deletion is chosen, the default data, method, sequence, and locked items will not be deleted.

3 Click **OK** to create the archive.

Procedure to restore an archive

To restore an archive:

1 Start **Data Analysis**.

   a On the desktop, double-click **Data Analysis**.

   b Log on.

2 Set up the restore.

   a Select the **Archive/Restore** menu.

   b Select a **Restore Archive** item.

   c Select the archive file to be restored. (The file must be on the hard disk.)

3 Click **OK** to restore the archive.

To View Audit Files

Audit trails

The system audit trail logs system activities and is created and maintained by the software. It records logins (both attempted and successful), running the MSD Security applications, data archival, and similar actions.

Run-related audit trails document the record creation, all modifications, and electronic signatures, and are associated with specific electronic records.

Method, sequence, and data audit files

Every electronic record created by the MSD Security ChemStation is associated with an audit file. The audit file is written by the software and records the creation, modification, and other changes to the electronic record. There are three types of audit files: Method, Sequence, and Results.

To view an audit file, open **Data Analysis** and select **Tools/View Audit File**. Audit files cannot be edited or deleted.

To print an audit file, right-click in the audit file window and select **Print** from the popup menu.

Event Viewer logs

The MSD Security ChemStation uses the Windows Event Log Service to record security and application events in the system audit trail. The logs can be viewed using the Event Viewer, which is accessed from the Control Panel **Administrative Tools** dialog box.

Security events

The Windows Security Event Log can record security events such as valid and invalid log on attempts, as well as events related to resource use, such as creating, opening, or deleting files. An administrator can specify what events are recorded in the security log. For example, if you have enabled log on auditing, attempts to log on to the system are recorded in the security log. The MSD Security ChemStation installation sets the default events that are recorded to be:

- Log on/Log off, success and failure
- Policy Change, success and failure
- Account Management, success and failure
- Account Log on, success and failure

For example, each time an MSD Security ChemStation user is successfully authenticated, there will be a log on event, a log off event, and an account log on event recorded in the security log. If an administrator changes the auditing policy or adds a new user, those events will be recorded. Unsuccessful log on or authentication attempts will also record events in the security log. The Security Event Log is a standard Windows Event Log.

Agilent MSD Security ChemStation events

The MSD Security ChemStation records application-specific events during normal operation in a custom event log named Agilent MSD Security ChemStation Log. These include program startup and shutdown, warnings, and error events at the application level such as changes to the current “user of record” of the applications.

Other events

Besides the Security Event Log, two other logs are standard with Windows, the Application Event Log and the System Event Log. They record events from other applications, and events of significance to the operating system, respectively.

Log file settings

At MSD Security ChemStation installation, the log sizes are set to be approximately 10 MB each and the retention period is set to 1 week. An administrator can change these values in the Event Viewer. The values should be set to reflect the backup frequency that you prefer; the defaults are for a weekly backup of the event logs.

Examine the logs frequently over the first few weeks of operation to see how fast they grow in your environment, then adjust the log sizes to be somewhat larger (for example, 25%) than the growth during the retention (backup) period you choose. Then, when you backup at the chosen retention interval, the saved log files will have slightly overlapping entries, to be sure that none are missed.

Event Logs Backup (XP)

Backup

To schedule regular backups of the event logs, use the Windows Backup facility. The files that need to be backed up are in **%SystemRoot%\Config**, where **%SystemRoot%** represents the name of the directory where the Windows operating system is installed, usually C:\Windows.

Log on to Windows as the local Administrator to set up the backup schedule for event logs.

The files to be backed up are listed in Table 4.

Table 4 Event logs to be backed up

File Name	Description
AppEvent.Evt	Windows application event log
SecEvent.Evt	Windows security event log
SysEvent.Evt	Windows system event log
AgtMSDSC.Evt	Agilent MSD Security ChemStation log (custom)

To start the backup utility and set up a scheduled backup:

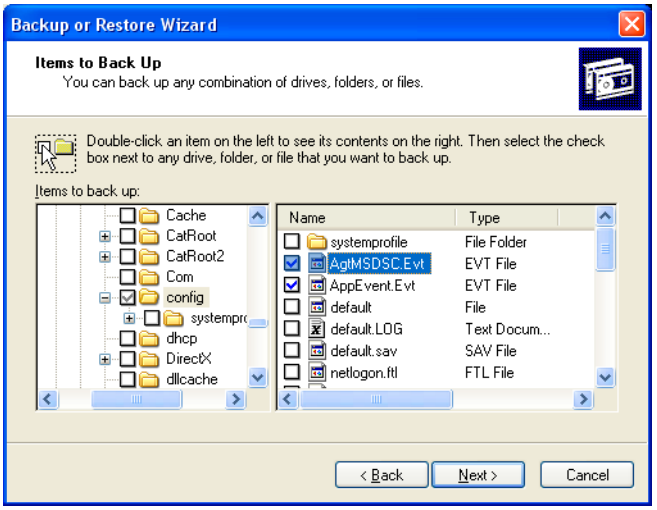
1 Log on to Windows as a Window Administrator or other user with administrative privileges.

2 Start the **Backup or Restore Wizard**.

   a Execute **Start/All Programs/Accessories/System Tools/Backup** to see the **Welcome** screen.

   b Click **Next** to continue. The **Backup or Restore** screen appears. Select **Backup files and settings** and click **Next**. The **What to Back Up** screen appears.

c Select **Let me choose what to back up** and click **Next**. The **Items to Back Up** screen appears. (The other choices are not appropriate to event log backup.)



3 Select the files to back up.

a Browse to %System Root%\Config and select the files listed in Table 4 on page 53. (%System Root% is usually C:\Windows.)

b When all files are selected, click **Next**. The **Backup Type**, **Destination**, and **Name** screen appears.

4 Select the backup destination.

Select a listed device or **Let me choose a location not listed here**. Click **Next**. The **Let me choose...** selection allows you to create and backup to a new file on the hard drive.

5 Name the backup file (if you selected **Let me choose...**).

a The **Save As** screen appears. Indicate where the file should be saved and supply a file name.

b Click **Save**. The **Completing the Backup or Restore Wizard** screen appears.

6 Specify the backup type.

a Click **Advanced**. The **Type of Backup** screen appears. (If you click **Finish**, the backup will be performed using the existing settings. **Advanced** allows you to make additional settings.)

b Select **Normal** and click **Next**. The **How to Back Up** screen appears. (Normal backs up the files and marks them as such.)

7 Verify data, if desired (recommended).

a Select **Verify data after backup**. (Verify will perform the backup, then read the backed up data and compare it with the original files.)

b Click **Next**. The **Backup Options** screen appears.

8 Select how to treat existing backups.

a Select either

- **Append this backup to the existing backups** (For backup locations on the system disk, Append is usually appropriate.)

  OR

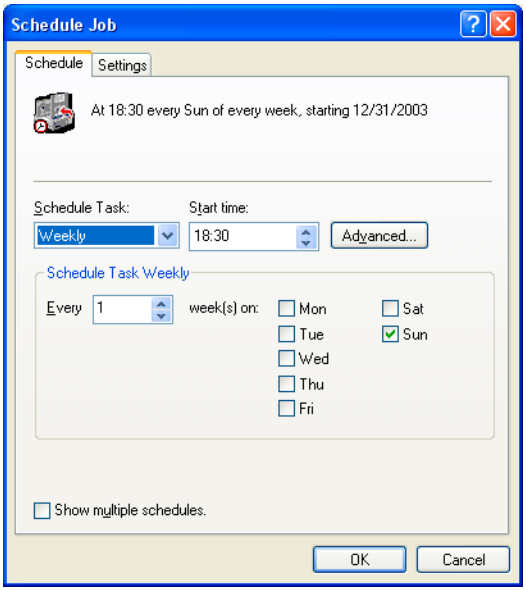
- **Replace the existing backups**

b Click **Next**. The **When to Back Up** screen appears.

9 Schedule automatic backups. You may want to wait until you know how fast the files grow (see page 52) before setting up a schedule.

a Select **Later**.

b Enter a **Job Name** and click **Set Schedule**. The **Schedule Job** screen appears.



c Select an appropriate schedule.

d Select the **Settings** tab, and choose options that apply to your situation.

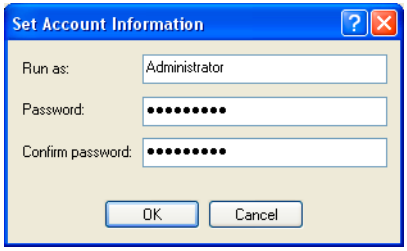
e Click **OK** when finished. The **Set Account Information** screen appears.

10 Select the user who will make the backup. (The user must have access to the files to be backed up. The local Administrator account is usually appropriate.)

a Enter the user name in **Run as**:

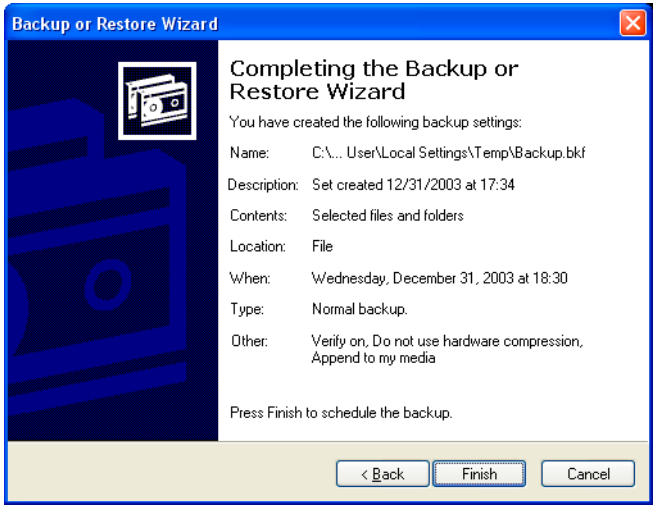
b Enter and confirm the password.

c Click **OK**.



11 Perform the backup.

You have reached the end of the **Backup or Restore Wizard**. Verify that the information shown is correct and then click **Finish**. Your scheduled backup will be created.


Backup or Restore Wizard
Completing the Backup or Restore Wizard
You have created the following backup settings:
Name: C:\... User\Local Settings\Temp\Backup.bkf
Description: Set created 12/31/2003 at 17:34
Contents: Selected files and folders
Location: File
When: Wednesday, December 31, 2003 at 18:30
Type: Normal backup.
Other: Verify on, Do not use hardware compression, Append to my media
Press Finish to schedule the backup.
< Back
Finish
Cancel

Restore

A similar wizard guides you through the restore process. The key to restoring these event logs is to ***restore to an alternate location***, so the old archives do not overwrite the current logs.

For Windows XP®, selected wizard dialogs are shown here.

1 After selecting **Restore files and settings**, click **Next**. The **What to Restore** screen appears.

2 Select the files to restore and click **Next**. The **Completing the Backup or Restore Wizard** screen appears.

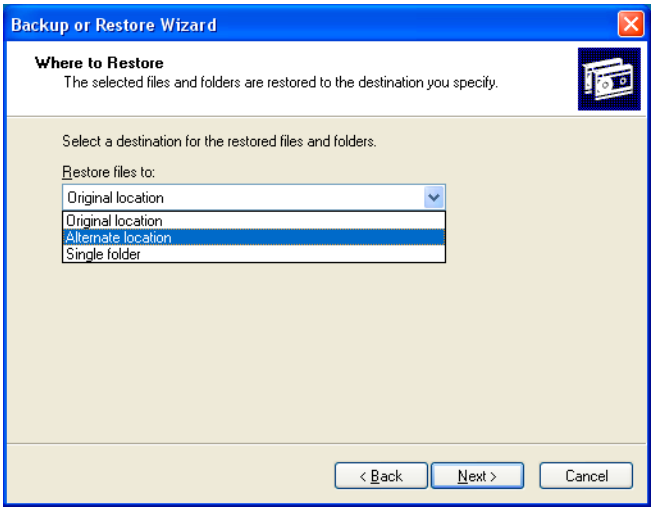
3 Click **Advanced...** to open the **Where to Restore** screen.

Backup or Restore Wizard
Completing the Backup or Restore Wizard
You have created the following restore settings:
Restore from: Backup.bkf created 12/31/2003 at 19:01
Type: File
Restore to: Original locations
Existing files: Do not replace
To close this wizard and start the restore, click Finish.
To specify additional restore options, click Advanced.
Advanced...
< Back
Finish
Cancel

4 Select one of the following:

- **Original location** overwrites the active log file with the contents of the archive. You probably do *not* want to do this because it destroys current information.
- **Alternate location** allows you to select a directory and restores the entire backup file tree using that directory as the root. It does not alter the active log files.
- **Single folder** is similar to **Alternate location**, except that it does not maintain the file tree; all files are placed in the root directory.

For most purposes, you will want **Alternate location**.


Backup or Restore Wizard
Where to Restore
The selected files and folders are restored to the destination you specify.
Select a destination for the restored files and folders.
Restore files to:
Original location
Original location
Alternate location
Single folder
< Back
Next >
Cancel

5 Click **Next** to continue with the wizard. The **How to Restore** screen appears.

6 Select **Leave existing files (Recommended)** and click **Next**. The **Advanced Restore Options** screen appears.

7 Select **Restore Security Settings** and **Preserve existing volume mount points**.

8 Click **Next**. You have reached the end of the **Backup or Restore Wizard**. Verify that the information shown is correct and then click **Finish**.

Maintenance Schedule

Detailed maintenance tasks are described in the hardware manuals supplied with your system. How often you need to perform system maintenance may vary for your system. Keep a maintenance record.

Every day

- ❑ Check, and if necessary, replace the septum.
- ❑ Check the tightness of the injection port liners.
- ❑ Check the tightness of the column nuts.

Every week

- ❑ Check the foreline pump fluid level.
- ❑ Change the injection port liners and O-rings.

Every month

- ❑ Clean the split/splitless inlet vent line trap.
- ❑ Check for leaks (inlet, column connections).

Every 3 months

- ❑ Replace gas cylinders (when below 500 psig).

Every 6 months

- ❑ Replace the foreline pump fluid.
- ❑ Check, and if necessary, refill the calibration vial.

Every year

- ❑ Check, and if necessary, replace the diffusion pump fluid.
- ❑ Recondition or replace internal and external traps and chemical filters on the GC.

As needed

- ❏ Tune the MSD.
- ❏ Clean the ion source.
- ❏ Replace the carrier gas trap.
- ❏ Replace worn out parts (filaments, EM, etc.).
- ❏ Replace the column.
- ❏ Lubricate seals.

Safety Warnings

WARNING

Do not perform maintenance with the MSD on or connected to its power source unless specifically instructed to by documentation supplied with the MSD.

The GC/MSD interface can be on and at a dangerously high temperature even though the MSD is off. After it is turned off, the GC/MSD interface cools very slowly. Make sure all parts have cooled before handling them.

Be careful when working behind the GC. During cool-down cycles, the GC will emit hot exhaust that could cause burns.

The oil trap provided for your foreline pump stops only foreline pump oil. If you are analyzing toxic chemicals or using toxic solvents, remove the oil trap and use a hose to route the foreline pump exhaust out of your laboratory.

Use chemical-resistant gloves and safety glasses when replacing pump fluid. Avoid all contact with the fluid.

The insulation around the inlets, detectors, valve box, and insulation cups is made of refractory ceramic fibers (RCF). Avoid inhalation of RCF particles. Ventilate your work area, wear long sleeves, gloves, safety glasses, and a disposable respirator. Dispose of insulation in a sealed plastic bag. Wash your hands with soap and cold water after handling RCFs.

6
Tips

Operating Tips 64
Error Messages and Troubleshooting 66
Error Messages 66
Troubleshooting Tips 67

This chapter is a collection of assorted facts about system operation and problems.

Operating Tips

- Back up your data and methods **regularly** to avoid loss of data if the files are accidentally overwritten or deleted, or if a hardware problem develops with your disk drive.
- Make sure the tune file you are using is appropriate for your samples.
- Save Tune reports in a notebook for future reference.
- Perform system maintenance as indicated by the maintenance schedule in the GC and MSD hardware documentation. Keep a record of all maintenance performed.
- When venting the MSD, take advantage of the cool GC to do maintenance such as replacing inlet liners, septa, etc.
- After pumpdown, wait *at least 2 hours* for the MSD to reach thermal equilibrium before tuning or acquiring data.
- Optimum sensitivity generally occurs at column flow rates of 1.2 µL/minute or less.
- When injecting volumes greater than 1 µL, use the pulsed splitless mode and increase the initial oven temperature 10–20°C.
- For splitless injections, pulsed splitless mode gives more quantitative sample transfer onto the column. A pulse pressure of twice the initial inlet pressure is typical.
- Selecting Constant Flow mode will provide the most efficient separation in most cases. It also results in constant sensitivity throughout the temperature program.
- For a new column, check that the column nuts are still tight after the first few oven temperature cycles.
- Use the [Config Status] keys on the 6890 GC keypad to set the three display items most important to you (time remaining, oven temp, etc.). These are then always visible regardless of which GC/MSD Security ChemStation view is on top.
- Rinse and refill autosampler wash vials. Do not add more solvent to a partially full vial.

- Use the following table as a guide to using the Selected Ion Monitoring (SIM) or Scan acquisition modes.

Task	Mode
Analyze a mixture with unknown components.	Scan, or Scan and SIM
Analyze a mixture with known components in unknown amounts (quantitate).	Scan, or SIM, or Scan and SIM
Identify the presence of a few known compounds at low levels within a mixture.	SIM

- When choosing masses for SIM, use the exact mass printed in the Tabulation report, not the nominal mass annotated on the spectrum display. This provides more accurate data.
- When doing SIM analysis, use low resolution mode unless you are trying to determine the ratios of masses 1 μ apart. Low resolution provides maximum sensitivity and repeatability.
- Choose the narrowest scan range that still produces good library search results. This allows more spectra across the peak and better quantitation.

Error Messages and Troubleshooting

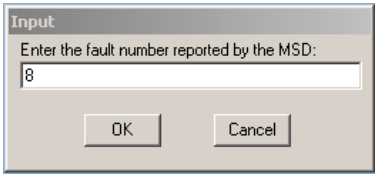
Error Messages

Sometimes a problem in your MSD will cause an error message to appear in the GC/MSD Security ChemStation software. Some error messages appear only during tuning. Other messages appear during tuning or instrument control.

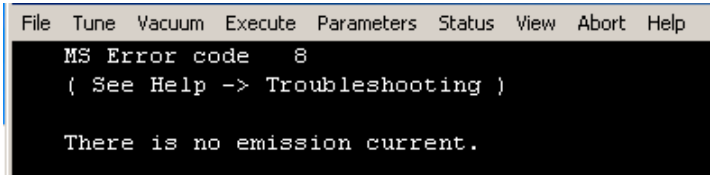
Sometimes, instead of a message, only a number will appear. This number can represent one or more error messages.

To translate a number into an error message:

1. Note the number.
2. In Instrument Control view, select **View/Tune and Vacuum Control**.
3. Select **Status/MS Error Codes**.
4. Type the error number in the box provided and click **OK**.



The corresponding error message(s) will be displayed.


File Tune Vacuum Execute Parameters Status View Abort Help
MS Error code 8
(See Help -> Troubleshooting)
There is no emission current.

Troubleshooting Tips

MSD LAN error

MSD is on, but status flashing "Server not found! Check LAN connection"

This is normal when the MSD is initially turned on. It means the GC/MSD Security ChemStation has not yet established contact with the MSD. If the flashing continues after the pumpdown is initiated, one or more of the following occurred:

- Temporary power failure interrupted communications.
- Bad connection between the MSD and the GC/MSD Security ChemStation and/or the Agilent Bootp service and/or the switch/hub.
- MAC and IP addresses for the MSD are not properly configured in the Agilent Bootp service for the LAN.

Rising baseline

- Column bleed
- Other contamination

Foreline or vacuum manifold pressure too high

- Excessive column flow
- Air leak
- Diffusion pump fluid level too low
- Diffusion pump fluid is contaminated
- Foreline pump oil level too low
- Foreline pump oil is contaminated
- Constricted foreline hose (this would cause the vacuum manifold pressure to be too high but the foreline pressure to be too low)

High background in mass spectra

- Air leak
- Foreline or vacuum manifold pressure too high
- Other contamination

Ions at *m/z* 18, 28, 32, and 44

- Detector vented recently (residual air and water)
- Air leak

Isotopes missing or isotope ratios incorrect

- Incorrect tuning
- Dirty ion source
- High background
- Electron multiplier voltage too high
- Repeller voltage too high
- High scan speed (Scan mode)
- Low dwell time (SIM mode)
- Peaks too wide or too narrow
- Repeller and ion focus leads have been reversed

No peaks

- Incorrect sample concentration
- No analytes present
- Syringe missing or not installed correctly (ALS only)
- Empty sample vial
- Injection in split mode instead of splitless mode

Tailing peaks

- Active sites in sample path
- Injection too large
- Injection port too cool
- Column flow too low
- GC/MSD interface or ion source too cool

Peaks with flat tops

- Solvent delay time too short
- Display scale is wrong
- Injection too large
- Electron multiplier voltage too high

Peaks with split tops

- Bad injection technique
- Injection too large

Inconsistent peakwidths

- Incorrect tuning
- No PFTBA in calibration vial
- Calibration valve failure
- Dirty ion source
- Worn out electron multiplier
- MSD has not had enough time to reach thermal equilibrium
- Large variations in the temperature of the lab

Poor repeatability

- Dirty syringe needle
- Leaking injection port
- Mismatched injection port liner and injection size
- Loose column connections
- Variations in pressure, column flow, and temperature
- Dirty ion source
- Loose connections in the analyzer
- Ground loop

Poor sensitivity

- Incorrect tuning
- Tune file does not match type of analysis
- Incorrect temperatures
- Incorrect sample concentration
- Leaking injection port
- Incorrect split ratio
- Purge off time in splitless mode too short
- Excessive pressure in the MSD
- Dirty ion source
- Air leak
- Detector is not working correctly
- Poor filament operation
- Incorrect mass filter polarity

Retention time (RT) drift

- Column has been shortened (shorter RT)
- Old column (shorter RT)
- Active sites in sample path (longer RT)
- Reduced column flow (longer RT)
- Injection port leak (longer RT)
- Initial oven temperature changed (up = shorter RT, down = longer RT)

Refer to the Troubleshooting the MSD section of the online Help for more detailed information.

6 Tips

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